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ANALYTICAL REFERENCE SERVICE. REPORT ON SAMPLE TYPE I

Robert A. Taft Sanitary Engineering Center
Cincinnati, Ohio

2 July 1956

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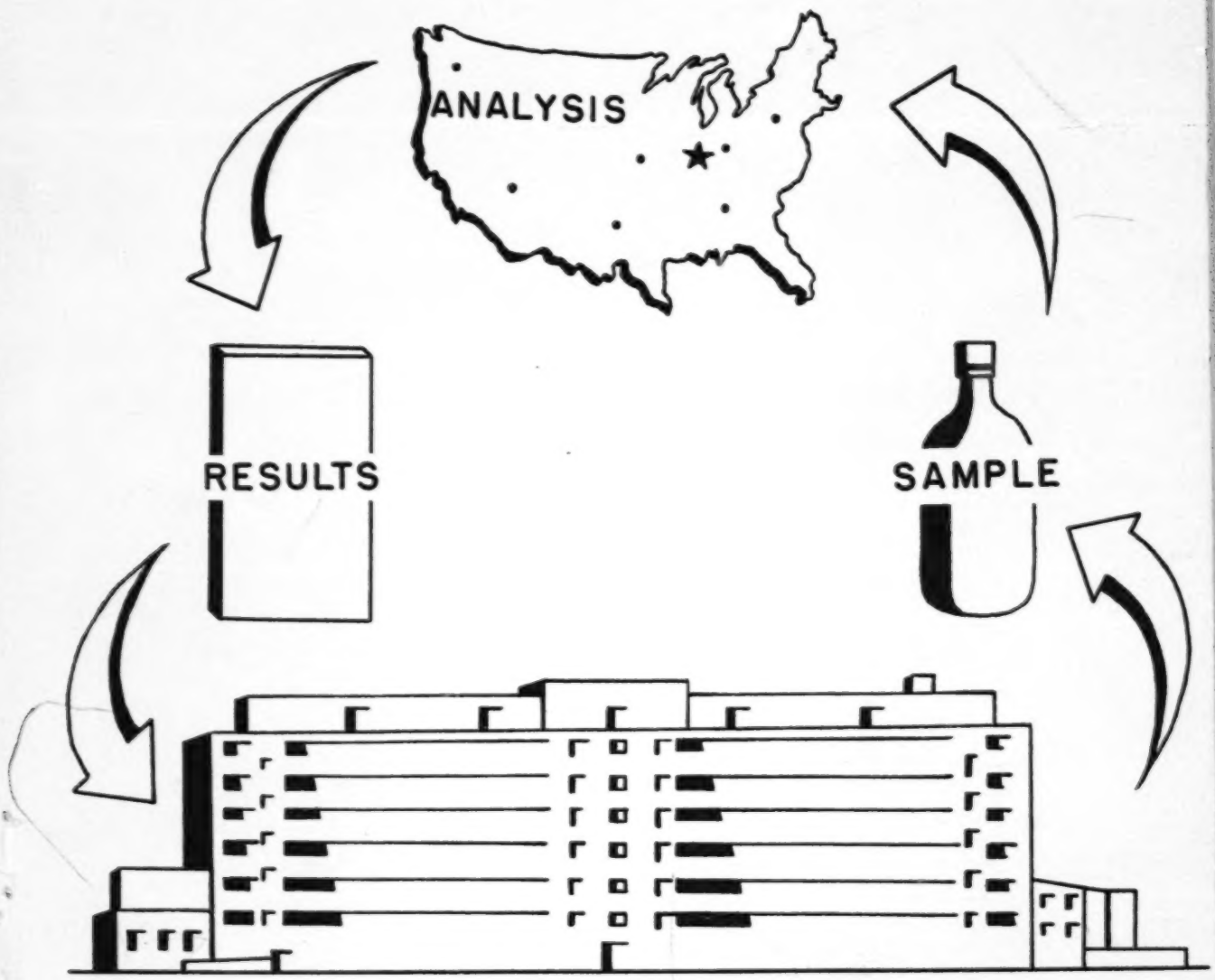

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(Distributed March 1955)

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Montana State Board of Health
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New Mexico Dept. of P. H.
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Vermont Water Conservation Board
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West Virginia State Water Commission

Federal Agencies

Army Chemical Center, Edgewood, Maryland
Sanitary Engineering Division, 13th Naval District
U. S. Geological Survey, Department of the Interior
Columbus, Ohio
Sacramento, California
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Universities

Department of Sanitation, Rutgers University

ANALYTICAL REFERENCE SERVICE

INTRODUCTION

The Analytical Reference Service of the Robert A. Taft Sanitary Engineering Center has as its primary goal the evaluation of laboratory methods in the field of sanitary engineering through the cooperative efforts of organizations having major responsibility for laboratory programs, chiefly state and federal agencies. The exchange of information in addition to the actual experience gained in this type of activity has in the past led to increased proficiency of the participants.

It is believed that through this service a reliable means of evaluation of old and new analytical methods is provided. Other benefits are these:

a. A statistical evaluation of analytical procedures as practiced by similar laboratories using identical samples on a nation-wide scale.

b. Exchange of information regarding weaknesses of currently approved methods.

c. An indication to interested research laboratories of methods requiring further development or new approaches.

d. Opportunity for a participating laboratory to evaluate its own procedures and to revise those methods requiring improvement.

PLAN OF OPERATION

Initially, efforts are being directed towards the chemical examination of water. The scope of the program may be broadened gradually to include other types of examinations.

Samples containing measured amounts of the desired constituent are prepared using the most refined grade of chemicals available. Decisions as to the qualitative makeup of any particular sample follow suggestions from the member groups and from the Sanitary Engineering Center.

Following preparation of a sample, a notice with a self-addressed reply card is sent to member laboratories announcing the availability and qualitative makeup of the sample. An affirmative reply institutes immediate shipment of a portion of the particular sample, for which a polyethylene container is used. Results are requested within some reasonable period, usually sixty days.

REPORTING OF RESULTS AND COMMENTS

Data are returned to the Sanitary Engineering Center on forms supplied with the sample. Space is provided on the forms for numerical values and a narrative critique of the analytical methods employed.

The values found by the cooperating laboratories are compiled as in the accompanying report. From examination of the combined data, it is hoped that reliable figures relating to accuracy and precision of the methods for the various materials may be obtained. Obviously, the larger the number of laboratories cooperating in a survey, the more reliable the statistical values will be.

It is also strongly desired that all laboratories supply comments on the analytical methods they use. A complete log of pertinent details, difficulties, modifications, sources of error, and other factors would be extremely valuable. Such a compilation of comments could represent an exchange of ideas and opinions that could well become a major part of the final report.

REPORT ON SAMPLE TYPE I

PREPARATION

The substrate for Sample Type I consisted of the equivalent of double-distilled water, i.e., distilled water put through a de-ionizing column. Accurate volumes of standardized solutions of calcium chloride, magnesium sulfate, sodium nitrite, potassium nitrate, and sodium bicarbonate were added to the substrate to furnish the desired concentrations.

The determinations requested for this sample were:

Calcium	Nitrate nitrogen
Magnesium	Total alkalinity
Chloride	Total hardness
Sulfate	Sodium (optional)
Nitrite nitrogen	Potassium (optional)

COMMENTS ON RESULTS

General

A large section of this report consists of the actual data as received from the participants, along with the evaluations and comments. However, bar graphs plotted as "per cent deviation from amount added" have been included for each analysis and laboratory. The use of the graphs serves a two-fold purpose:

1. Each cooperating laboratory, knowing its own code number, may judge how the accuracy of its work compares to that of other laboratories.

2. The overall accuracy of methods for any constituent may easily be judged.

It should be remember that "per cent error" evaluation shows a valid relationship on the same ion, but not on different ions. For example, an error of .01 mg/l in the nitrite determination will result in a 6.7% error, while an equivalent error in sulfate is negligible.

A casual examination of the bar graphs reveals a low result on calcium from all reporting laboratories. It is suspected that a loss of calcium by precipitation occurred in the preparation of the sample. A similar phenomenon is exhibited in the chloride data, all results being in excess of the "amount added". In this case, the occurrence of chlorides as impurities in other reagents is undoubted responsible for the excess amounts detected. However, since the deviation between laboratories for both calcium and chloride was very small, and since the deviation from the amount added can be logically explained, it is felt that these materials can be accurately measured. The consistency of data with regard to sulfate, hardness, and magnesium indicates that these may also be accurately measured.

The measurement of alkalinity was originally considered to be of dubious value, since the alkalinity is influenced by absorption of carbon dioxide from air. However, since the actual determination involves only a simple titration it was included.

Sodium and potassium determinations were made optional. The amounts added were not specifically chosen but were the result of reagent additions for other ions. It is obvious from the reported results that more sodium was added than can be accounted for by calculation. Again, sodium, like chloride, is a common impurity in most reagents and can easily be picked up from laboratory glassware.

Specific

Calcium

Two principal methods were reported for use in calcium determination, oxalate-permanganate and Versenate. Neither method shows a pronounced statistical superiority over the other, but the Versenate method seems much preferred because of its simplicity. A single laboratory reported calcium by calculation, while another used the gravimetric method. Both results compare very favorably with the overall average.

Nitrite

The alpha-naphthalamine method apparently is the only method for nitrite measurement in current use. In view of the low concentration, the large majority of reported measurements were extremely good. Of seventeen laboratories reporting, four had perfect analyses. Since the results showed random distribution, deviations from the amount added are probably due to usual technical errors - improperly cleaned glassware, erroneous standards, fading of color in samples, etc. In general, those laboratories which performed well on other analyses showed good results on the nitrite determination.

Nitrates

Of eighteen reported values for nitrates, sixteen were obtained using the phenoldisulfonic acid method. The results were almost uniformly low, but re-examination of the procedures for sample preparation revealed no detectable errors. The failure to obtain results consistent with the amount added can only be ascribed to a weakness of the method itself. The failure of the method may be due to lack of, or inadequacy of, correction for chlorides which are known to interfere. This explanation, however, cannot be definitely claimed because of insufficient information in the comments.

It is interesting to note that two of the laboratories which performed well on other analyses reported perfect results on this analysis. Both of these laboratories also report the use of ammonium hydroxide in place of sodium hydroxide for alkalinization of the acid reagent.

Sulfate

Seventeen laboratories report sulfate analyses based on gravimetric techniques. The average per cent deviation from the amount added is 2.5, which figure also includes data based on volumetric and turbidimetric procedures. Obviously the gravimetric procedure is entirely

capable of yielding satisfactory results, although the consensus of opinion is that the work is time-consuming and tedious. Calcium does not appear as a serious interference.

Sulfate determinations based on turbidimetric methods also yielded good results. Since only two laboratories reported use of tetrahydroxyquinone, no conclusions can be drawn regarding the method.

Alkalinity

The reported values for alkalinity show wide variations despite the fact that the procedure is based on simple volumetric techniques. However, it has long been known that the use of methyl orange offers opportunity for personal error. The endpoint error is demonstrated graphically by the wide and random variation in results.

In the electrometric or pH methods, which eliminate subjective errors of color change, the results from five laboratories are very good. This method would be recommended for any laboratory possessing a pH meter.

Hardness

The titration of hardness salts, with ethylene diamine tetraacetic acid (reported here by the trade name "Versenate") has been very widely accepted by sanitary chemists in the past few years. Illustration of the results show the method to be very acceptable and reliable, regardless of the indicator used.

Magnesium

In the Summary of Results, it is seen that for magnesium the average deviation from amount added is only 5.4%. This average is based on three totally different methods, including gravimetric, volumetric, and calculation.

The results reported by gravimetric procedure are very close to the amount added, with one exception. The results obtained by calculation also show an average very close to the amount added, but the deviation for this method shows wider variance. Three laboratories reported results by "Versene" titration, and, while the results appear acceptable, this method did not serve as well as the gravimetric procedure.

Chloride

All laboratories reported chloride results higher than the amount added, although the deviation between reported values was small. If the arithmetic mean of 223 ppm is arbitrarily chosen as the true value of chloride in the sample, then 23 of the 24 reported values are within 10% of the true value, 16 results are within 5%, and 11 results are within 2%, regardless of the method used.

In the light of such replication between laboratories, it can be assumed that methods for the measurement of chloride ion are reliable at this concentration. The Mohr method is by far the most popular reported, but the mercuric nitrate method also shows excellent results. Laboratories using the mercuric nitrate method report a sharper endpoint than is obtained with the other volumetric procedures.

Sodium and Potassium

These analyses were made optional because it was recognized that only a few laboratories would be willing to make the attempt. It was also anticipated that unless very special precautions were taken, contamination of the sample by sodium would occur. The results reported for sodium are fairly uniform although they differ from the amount added. The difference, of course, is sodium contamination.

One fact is prominent in the Na-K analyses. All laboratories used the flame technique while the more tedious gravimetric methods were ignored. In view of the increasing demand for sodium and potassium analyses it seems highly desirable that a Standard Method, employing flame technique be devised. However, even lacking a standard procedure the inter-laboratory results are in good agreement.

SUMMARY OF RESULTS

CALCIUM

Reported as Ca. Amount Added = 110 ppm.

Lab Number	Replicate	Average	Average Deviation Within Each Lab. ppm	Average Deviation Within Each Lab. per cent	Average Deviation From Amount Added ppm	Average Deviation From Amount Added per cent	Method
SEC	94 96 95	95	0.7	0.7	-15	-14	Oxalate-permanganate
1	97.0 97.8 97.8	98*	0.3	0.3	-12	-11	Oxalate-permanganate
2	109 106.6 106.6	107*	1.1	1.0	- 3	- 3	Oxalate-permanganate
	107.9	108*	--	--	- 2	- 2	Versenate
3	100 106 106	104	2.7	2.6	- 6	- 5	Versenate
4	100 100 96	99	1.8	1.8	-11	-10	Versenate
5	90 90 90	90	0	0	-20	-18	Versenate-(murexide)
6	96.6 97.1 96.6	97*	0.2	0.2	-13	-12	Oxalate-permanganate
7	98 96 97	97	0.7	0.7	-13	-12	Versenate (ammonium purpurate)
8	82 79 82	81	1.3	1.6	-29	-26	Versenate
9	92 93 --	93	0.5	0.5	-17	-15	Versenate (Betz solns. and indicator)

10	96 97 97	97	0.4	0.4	-13	-12	Versenate (murexide)
	90 90 91	90	0.4	0.4	-20	-18	Oxalate-permanganate
11	91.3 91.5 91.4	91*	0	0	-19	-17	Versenate (calcium indicator capsules)
	98	98	--	--	-12	-11	Oxalate-permanganate
12	98.3 98.8	98*	0.2	0.2	-12	-11	Oxalate-permanganate (1 precipitation)
	96.3 96.3	96*	0	0	-14	-13	Oxalate-permanganate (2 precipitations)
	97.7 92.2	95*	2.7	2.8	-15	-14	Calculation
13	98 98 98	98	0	0	-12	-11	Versenate (ammonium purpurate)
14	108.2 102.2 104.2	105*	2.2	2.1	- 5	- 4	Oxalate-permanganate
15	98.2 97.8 101.6	99*	1.6	1.6	-11	-10	Oxalate-permanganate
16	104 104 105	104	0.4	0.4	- 6	- 5	Oxalate-permanganate
17	96 97 97	97	0.4	0.4	-13	-12	Versenate (ammonium purpurate)
18	108 106 109	108	1.1	1.1	- 2	- 2	Versenate
19	98 98 97	98	0.4	0.4	-12	-11	Versenate (ammonium purpurate)
20	101 102 --	101	0.5	0.5	- 9	- 8	Gravimetric

* Denotes rounded off value

COMMENTS ON CALCIUM DETERMINATION

Lab. 1:	No comments made.
Lab. 2:	Versenate method is used only when a quick determination of calcium for private samples is requested. It is thought to be sufficiently accurate for all practical purposes. The end point is not very sharp. The 0.1 N $KMnO_4$ method seems to be lacking in accuracy to a slight degree in that too little or too much washing may give too much or too little calcium. Too little washing leaves a little ammonium oxalate which is determined as calcium. Too much washing dissolves some of the calcium and results are too low. It is too much of a guess as to knowing the exact place to stop.
Lab. 3:	No comments made.
Lab. 4:	No comments made.
Lab. 5:	Less precise but satisfactory and with our limited experience we have not as yet come upon unusual interference. Time requirements and manipulation are at a minimum.
Lab. 6:	No comments made.
Lab. 7:	No comments made.
Lab. 8:	Method seems very good.
Lab. 9:	Method considered satisfactory.
Lab. 10:	Versenate - Direct titration with preliminary precipitation not needed, thus requires less time. Oxalate permanganate - Precipitation and redissolving are time consuming.
Lab. 11:	Versenate - Method regularly used. Method seems more rapid than APHA directions and eliminates several possibilities for technical errors. Oxalate-permanganate - Time consuming.

Lab. 12: It is the practice of this lab to report all calcium determinations by calculation except when a precise analysis of this element is demanded, whereupon the oxalate method is used. Determination of calcium by oxalate, although more exact, is too cumbersome and time consuming to justify its use as a regular routine procedure, when the results achieved by calculation are generally satisfactory.

The method of calculation is merely computing the difference between the total hardness and the magnesium as calcium carbonate. We find this procedure more expedient because it is possible to determine magnesium rapidly by a photometric method.

Lab. 13: End point not as distinct as in the hardness test, but reproducible results, varying by only 0.1 ml. are obtained.

Lab. 14: No comments made.

Lab. 15: No comments made.

Lab. 16: Instead of digesting the calcium oxalate in alkaline medium, we use a slightly acid solution (acid to methyl red) and seem to obtain better results. This method is to be criticized, of course, in that the total time for precipitation, digestion, filtration, is great.

Lab. 17: Rapid; easily done; results highly satisfactory.

Lab. 18: Method accurate enough for routine purposes.

Lab. 19: No comments made.

Lab. 20: No comments made.

NITRITE

Reported as N. Amount Added = 0.15 ppm.

Lab. Number	Replicates	Average	Average Deviation Within Each Lab ppm	Average Deviation Within Each Lab per cent	Average Deviation from Amount Added ppm	Average Deviation from Amount Added per cent	Method
SEC	0.17 0.17 0.17	0.17	0	0	+0.02	+13	Alpha-naphthylamine
1	0.12 0.12 0.12	0.12	0	0	-0.03	-20	Alpha-naphthylamine
3	0.15* 0.20* 0.20*	0.18	0.022	12	+0.03	+20	Alpha-naphthylamine
4	0.10 0.15 0.15	0.13	0.022	17	-0.02	-13	Alpha-naphthylamine
5	0.15* 0.15* 0.14*	0.15	0.002	1	0	0	Alpha-naphthylamine
6	0.16 0.16 0.16	0.16	0	0	+0.01	+7	Alpha-naphthylamine
8	0.10 0.10 0.09	0.10	0.004	4	-0.05	-33	Alpha-naphthylamine
9	0.18 0.18 --	0.18	0	0	+0.03	+20	Alpha-naphthylamine
10	0.12 0.125 --	0.12	0.002	2	-0.03	-20	Alpha-naphthylamine

11	0.095 0.095 0.095	0.095	0	0	-0.05	-33	Alpha-naphthylamine
12	0.11 0.11 0.11	0.11	0	0	-0.04	-27	dimethyl alpha-naphthyl-amine
13	0.15 -- --	0.15	--	--	0	0	Alpha-naphthylamine
14	0.14 0.14 0.14	0.14	0	0	-0.01	-7	Alpha-naphthylamine
15	0.14* 0.15* 0.15*	0.15*	0.004	3	0	0	Alpha-naphthylamine
16	0.15 0.15 0.14	0.15	0.004	3	0	0	Alpha-naphthylamine
17	0.38 0.38 0.38	0.38	0	0	+0.23	+153	Alpha-naphthylamine
20	0.81 0.81 --	0.81	0	0	+0.66	+440	Alpha-naphthylamine

* Denotes rounded off value.

COMMENTS ON NITRITE DETERMINATION

Laboratory 1: No comments made.

Laboratory 3: No comments made.

Laboratory 4: No comments made.

Laboratory 5: Difficulty in obtaining dependable standard material. Silver nitrite is time consuming to prepare, and commercial nitrites may or may not be sufficiently pure.

Laboratory 6: No comments made.

Laboratory 8: Method appears satisfactory.

Laboratory 9: No comments made.

Laboratory 10: Not routinely done because samples are most often not fresh.

Laboratory 11: Difficulty is encountered in matching by visual methods.

Laboratory 12: Method used is that suggested by Snell (Colorimetric Methods of Analysis, D. Van Nostrand, 1939). The reagents employed are dimethyl alpha-naphthylamine in 4 N acetic acid in 95% methanol and a sulfanilic acetic acid mixture. The concentration of nitrite is determined photometrically. The color produced with dimethyl alpha-naphthylamine is superior in intensity, stability and brilliance to that with alpha-naphthylamine. The reagent is stable for at least sixty days.

Laboratory 13: No comments made.

Laboratory 14: No comments made.

Laboratory 15: No comments made.

Laboratory 16: No comments made.

Laboratory 17: Very sensitive and rapid.

Laboratory 20: No comments made.

NITRATE

Reported as N. Amount added = 1.2 ppm.

Lab. Number	Replicates	Average	Average Deviation Within Each Lab ppm	Average Deviation Within Each Lab per cent	Average Deviation from Amount Added ppm	Average Deviation from Amount Added per cent	Method
SEC	1.1 1.0 0.9	1.0	0.07	7	-0.2	-17	Phenoldisulfonic Acid
1	0.75 0.75 0.72	0.7*	0.013	2	-0.46	-38	Phenoldisulfonic Acid
2	1.3 1.4 1.4	1.4*	0.04	3	+0.17	+14	Phenoldisulfonic Acid
3	0.70 0.70 0.70	0.7*	0	0	-0.50	-42	Phenoldisulfonic Acid
4	0.60 0.80 0.60	0.7*	0.09	13	-0.53	-44	Phenoldisulfonic Acid
5	0.67 0.67 0.68	0.7*	0.04	6	-0.53	-44	Phenoldisulfonic Acid
6	0.68 0.68 0.68	0.7*	0	0	-0.52	-43	Phenoldisulfonic Acid
7	1.0 1.0 1.0	1.0	0	0	-0.2	-17	Hellige aqua tester
9	0.8 0.8 --	0.8	0	0	-0.4	-33	Phenoldisulfonic Acid
10	0.6 0.6 0.6	0.6	0	0	-0.6	-50	Phenoldisulfonic Acid

11	0.5* 0.5* 0.5*	0.5*	0	0	-0.7	-58	Phenoldisulfonic Acid
12	1.2 1.2 1.2	1.2	0	0	0	0	Phenoldisulfonic Acid
13	1.1 1.2 1.3	1.2	0.07	6	0	0	Phenoldisulfonic Acid
14	0.70 1.00 1.00	0.9*	0.13	14	-0.3	-25	Phenoldisulfonic Acid
15	1.0 1.0 1.0	1.0	0	0	-0.2	-17	Phenoldisulfonic Acid
16	0.61 0.61 0.61	0.6*	0	0	-0.59	-49	Reduction Method
17	1.5 1.5 1.4	1.5*	0.04	3	+0.27	+22	Phenoldisulfonic Acid
19	1.3 1.4 1.7	1.5	0.16	11	+0.3	+25	Phenoldisulfonic Acid
20	1.4 1.4 --	1.4	0	0	+0.2	+17	Phenoldisulfonic Acid

* Denotes rounded off value.

COMMENTS ON NITRATE DETERMINATION

Laboratory 1:	No comments made.
Laboratory 2:	This is not very accurate. In this particular determination a small amount of color was lost in the filter paper. If I were reporting to a private individual, I would say that there was approximately 1.5 ppm.
Laboratory 3:	No comments made.
Laboratory 4:	No comments made.
Laboratory 5:	We have found that dilution of a sample too high to be read does not give results comparable to the value obtained by evaporating a smaller sample. Evaporation of a 5 ml. sample, for example, ordinarily gives values much greater than those obtained by diluting the color obtained from a 10 ml. sample 1:2. Time and manipulation in this procedure are less satisfactory than most of the others, but are not unreasonable for routine work.
Laboratory 6:	No comments made.
Laboratory 7:	Determined from residue of total dissolved solids determination. Color evaluation with Hellige aqua tester.
Laboratory 9:	No comments made.
Laboratory 10:	Objectionable evaporation to dryness and often a different hue is found in samples and standards. Not routinely done - occasionally done on request.

Laboratory 11: Methods of preparing sample for analysis seem cumbersome. Difficulty encountered in matching color by visual methods.

Laboratory 12: Our procedure is as recommended by Taras (Anal. Chem., Vol. 22, No. 8, p. 1020, 1950). This is essentially the same as that given in Standard Methods except that the total alkalinity of the sample is not neutralized, and the use of ammonium hydroxide is preferred to that of the alkali hydroxides. We find the procedure to be an excellent one in all respects. The substitution of ammonium hydroxide for sodium or potassium hydroxide prevents the formation of an off-color brownish hue to the solution, which is a result of alkali hydroxides peptizing small amounts of silver hydroxide.

Laboratory 13: Standard Methods is used, substituting concentrated ammonium hydroxide for sodium hydroxide, and reading on photometer at 440 μ.

Laboratory 14: No comments made.

Laboratory 15: No comments made.

Laboratory 16: Test is satisfactory if sufficient time for reduction is allowed (preferably overnight) but then the process becomes quite time consuming.

Laboratory 17: The chloride interference is quite high in this method and so results are not too accurate. The concentration of nitrate is usually small and the accuracy of the method is generally suitable for most waters.

Laboratory 19: No comments made.

Laboratory 20: No comments made.

SULFATE

Reported as SO_4. Amount Added = 336 ppm.

Lab Number	Replicates	Average	Average Deviation Within Each Lab. ppm	per cent	Deviation of Average from Amount Added ppm	per cent	Method
SEC	340 323 331	331	5.7	1.7	-5	-1.5	Gravimetric
1	337 333 338	336	2	0.6	0	0	Gravimetric
2	347* 348* 352*	349*	1.7	0.5	+13	+4	Gravimetric
3	318 339 324	327	8	2.4	-9	-3	Gravimetric
4	326 325 333	328	3.3	1.0	-8	-2	Gravimetric
5	195 196 200	197	2	1.0	-139	-413	Tetrahydroxyquinone
	334 341 338	338	2.4	0.7	+2	+0.6	Gravimetric
6	347* 348* 349*	348*	0.5	1.4	+12	+3.6	Gravimetric
7	354 354 354	354	0	0	+18	+5.4	Gravimetric
8	310 308 324	314	6.7	2.1	-22	-6.5	Turbidimetric
9	337* 331* 337*	335*	2.6	0.8	-1	-0.3	Gravimetric

10	340 340 341	340	0.4	0.1	+4	+1.2	Gravimetric
11	337* 337* 337*	337*	0.1	0.03	+1	+0.3	Gravimetric
12	349 348 347	348	0.67	0.2	+12	+3.6	Gravimetric
	300 300	300	0	0	-36	-10.7	Photonephelometric
13	350 340 349	346	4.2	1.2	+10	+3.0	Turbidimetric
14	361* 336* 354*	350	9.4	2.7	+14	+4.2	Gravimetric (Standard Methods)
	364*	364*	--	--	+28	+8.3	Gravimetric (Mallman Methods)
15	332* 331* 332*	332*	0.4	0.1	-4	-1.2	Gravimetric
16	340 336 342	339	2.2	0.6	+3	+0.9	Gravimetric
17	345 346 347	346	0.7	0.2	+10	+3.0	Gravimetric
18	339 347 350	345	4.2	1.2	+9	+2.7	Gravimetric
19	350 340 340	343	4.4	1.3	+7	+2.1	Turbidimetric
20	328 328 --	328	0	0	-12	-3.6	Tetrahydroxyquinone

* Denotes rounded off value.

COMMENTS ON SULFATE DETERMINATION

Laboratory 1: No comments made.

Laboratory 2: No comments made.

Laboratory 3: No comments made.

Laboratory 4: No comments made.

Laboratory 5: Considerable investigative work on the volumetric determination of sulfate using THQ indicates that while satisfactory results can be obtained on solutions containing only sulfate, it is not suited for routine work on a variety of samples. On this particular sample the endpoints were sharp and distinct although the results were low. The gravimetric method seems to be the only completely satisfactory method. Unfortunately it is very time consuming for routine analysis.

Laboratory 6: No comments made.

Laboratory 7: No comments made

Laboratory 8: The turbidimetric method is fair for low concentrations of sulfates. Due to a high dilution error, not as accurate for high concentrations. Prone to give slight personal errors.

Laboratory 9: Gravimetric is somewhat time consuming; otherwise satisfactory.

Laboratory 10: Gravimetric involves time consuming precipitation and ignition and is unsatisfactory for quantities found in our waters. Turbidimetric preferred.

Laboratory 11: Gravimetric method seems to give better results then any others tried in this laboratory.

Laboratory 12: The procedure used in this particular instance, as well as the procedure in general use in our laboratory for the determination of sulfate is the standard gravimetric procedure involving the use of barium chloride as the precipitating agent to a slightly acid portion of the sample. The method we use is that given in Standard Methods in which no mention is made of the possible interference of calcium in the determination of sulfate. Due to the large number of samples analyzed in our laboratory, we find it impractical, because of the time element, to remove calcium, thereby introducing some degree of inaccuracy into our results. We are of the opinion, however, that the degree of inaccuracy as a result of failure to remove calcium is not serious enough to warrant the time involved in its removal. Allowing for its principal shortcoming of time consumption and the possibility of some error through failure to remove calcium, we hold that this method will yield most

accurate results when compared to several other more rapid methods that we have attempted to use with little success, such as the benzidine hydrochloride and tetrahydroxyquinone methods. For instance, the benzidine hydrochloride method works well with some ranges of sulfate ion concentrations only.

At the present time we are engaged in investigating the possibilities of a photonephelometric micro determination of sulfate as described by Toennis and Bakay in Analytical Chemistry, 5, No. 1, (1953). We believe this method has potential, particularly where the emphasis is placed on a rapid and not too accurate determination of sulfate rather than a slow and precise one, instances not at all uncommon in our work. The notable failing thus far in this photonephetometric procedure appears to be the error introduced as a result of employing large arithmetical factors necessary because of the small amounts of sample subjected to analysis in view of the nature of the procedure. Also, the results obtained by this method appear to be low.

Laboratory 13: The use of a mechanical shaking device and close timing have insured reproducible results. I think the method superior to the THQ method.

Laboratory 14: No comments made.

Laboratory 15: No comments made.

Laboratory 16: Much time and manipulation are involved, yet in many cases the precision and accuracy of this method justify its use. Other tests such as benzidine and THQ have been investigated and temporarily set aside for lack of suitable results.

Laboratory 17: This method has inherent errors and standardization of the technique is necessary to obtain reproducible results. Results are satisfactory, but the time factor is quite large.

Laboratory 18: No comments made.

Laboratory 19: No comments made.

Laboratory 20: No comments made.

ALKALINITY

Reported as $CaCO_3$. Median value = 19 ppm

Lab Number	Replicates	Average	Average Deviation Within Each Lab ppm	Average Deviation Within Each Lab per cent	Average Deviation from Median Value ppm	Average Deviation from Median Value per cent	Method
SEC	27.0 27.7 27.4	27	0.2	0.8	+8	+42	Methyl orange
1	20 20 20	20	0	0	+1	+ 5	Electrometric
2	18 18 18	18	0	0	-1	- 5	Methyl orange
3	15.0 16.0 17.0	16	0.7	4	-3	-16	Methyl orange
4	15.0 15.0 17.0	16*	0.9	5.7	-3	-16	Methyl orange
5	11.46 11.23 11.70	11*	0.16	1.4	-8	-42	Methyl orange
6	21.1 21.1 20.6	21*	0.2	1.1	+2	+10	Methyl orange (HCl)
7	21 21 21	21	0	0	+2	+10	Methyl orange (0.1 N HCl)
8	30 28 26	28	1.3	0.5	+9	+47	Methyl orange
9	14.0 14.5 --	14*	0.25	1.7	-5	-26	Methyl purple
10	14 15 15	15	0.4	2.9	-4	-21	Methyl purple

11	17.0 17.6 17.5	17*	0.2	1.1	-2	-10	Methyl orange
	14.9 15.2 15.0	15*	0.1	0.7	-4	-21	Methyl purple
12	21.0 21.0 --	21*	0	0	+2	+10	pH meter (pH 3.92)
13	20 22 20	21	0.9	0.4	+2	+10	Methyl orange
14	18.0 18.0 18.0	18*	0	0	-1	- 5	Methyl orange
15	19.0 19.0 19.0	19*	0	0	0	0	pH meter
16	19 19 19	19	0	0	0	0	pH meter (pH 4.3)
17	20 20 20	20	0	0	+1	+ 5	pH meter (pH 4.5)
18	27 27 28	27	0.4	1.5	+8	+42	Methyl orange
19	32 32 32	32	0	0	+13	+68	Phenolphthalein
20	18 18 --	18	0	0	-1	- 5	Methyl orange

* Denotes rounded off value.

COMMENTS ON ALKALINITY DETERMINATION

Laboratory 1: No comments made.

Laboratory 2: No comments made.

Laboratory 3: No comments made.

Laboratory 4: No comments made.

Laboratory 5: Methyl orange isn't the best indicator, nor the easiest to use. Otherwise time and manipulation are good.

Laboratory 6: No comments made.

Laboratory 7: No comments made.

Laboratory 8: Method appears satisfactory.

Laboratory 9: Methyl orange endpoint is difficult; methyl purple readily determined.

Laboratory 10: Methyl orange does not give a distinct endpoint. Methyl purple more satisfactory and just as accurate.

Laboratory 11: Endpoint indefinite with methyl orange; distinct with methyl purple.

Laboratory 12: Rather than making use of methyl orange indicator, our laboratory employs a pH meter (Beckman model H-2) in total alkalinity determinations. We have determined by experimentation that the pH of the theoretical endpoint of the methyl orange alkalinity (total alkalinity) is 3.92. Therefore, instead of using methyl orange, we merely titrate the sample with 0.02 N acid, using the pH meter to determine the endpoint. This procedure, we have found, eliminates the difficulty so often encountered by the use of methyl orange in determining the endpoint in total alkalinity determinations.

Laboratory 13: No comments made.

Laboratory 14: No comments made.

Laboratory 15: No comments made.

Laboratory 16: Method makes it possible to determine pH and alkalinity in one step. There is no criticism of the method if the pH meter is properly standardized and the electrodes sensitized or replaced as necessary.

Laboratory 17: Method rapid and gives good results.

Laboratory 18: No comments made.

Laboratory 19: No comments made.

Laboratory 20: No comments made.

HARDNESS

Reported As $CaCO_3$. Median value = 604 ppm

Lab Number	Replicates	Average	Average Deviation Within Each Lab ppm	Average Deviation Within Each Lab per cent	Average Deviation from Median Value ppm	Average Deviation from Median Value per cent	Method
SEC	603 604 603	603	0.4	0.06	-1	-0.2	Versenate (Eriochrome black T)
1	605 605 605	605	0	0	+1	+0.2	Versenate
2	604 604 604	604	0	0	0	0	Versenate
3	700 700 620	673	39	5.8	+69	+11.4	Soap Method
	640 660 640	647	9.0	1.4	+43	+7.1	Versenate (UniVer)
4	600 600 590	597	4.4	0.7	-7	-1.2	Versenate (UniVer)
5	596 597 596	596	0.4	0.07	-8	-1.3	Versenate (Eriochrome black T)
6	597.8 601.8 599.8	600*	1.3	0.2	-4	-0.7	Calculated
	604.0 600.0 596.0	600*	2.7	0.4	-4	-0.7	Versenate
7	628 634 633	632	2.3	0.4	+28	+4.6	Versenate (Eriochrome black T)
8	506 504 510	507	2.3	0.5	-7	-1.2	Versenate (Betz procedure)
9	590 590 --	590	0	0	-14	-2.3	Versenate (Betz soln. & indicator)

10	608 610 612	610	1.3	0.2	+6	+1.0	Versenate (Pontachrome black TA)
11	606.2 606.0 605.8	606*	0.1	0.02	+2	+0.3	Versenate
	605.9	606*			+2	+0.3	Calculated
12	596.6 599.6 594.0 598.0	597*	1.8	0.3	-7	-1.2	Versenate (MonoVer)
13	600 610 610	607	4.4	0.7	+3	+0.5	Versenate (UniVer)
14	631.4 617.2 622.2	624*	5.2	0.8	+20	+3.3	Calculated
15	592.0 580.0 572.0	581*	7.1	1.2	-23	-3.8	Versenate
16	613 613 611	612	0.9	0.1	+8	+1.3	Versenate (Pontachrome black T or UniVer)
17	602 602 604	603	0.9	0.1	-1	-0.2	Versenate (Eriochrome black T)
18	612 612 616	613	1.8	0.3	+9	+1.5	Versenate (ammonia buffer procedure)
19	614 610 604	609	3.6	0.6	+5	+0.8	Versenate (Eriochrome black T)
20	606 606 --	606	0	0	+2	+0.3	Versenate

* Denotes rounded off value.

COMMENTS ON HARDNESS DETERMINATION

Laboratory 1: No comments made.

Laboratory 2: No comments made.

Laboratory 3: No comments made.

Laboratory 4: No comments made.

Laboratory 5: We found that the solution recommended by Goetz, Loomis, and Diehl, containing magnesium chloride was preferable to the plain versenate solution. We also found that indicator made up in a basic solution (diethanolamine) is more satisfactory than when made up in isopropyl alcohol. We occasionally run into samples which will not give a satisfactory endpoint even when diluted. With these samples the indicator color is a dirty khaki color, and the change to blue is gradual and indeterminate.

Laboratory 6: No comments made.

Laboratory 7: No comments made.

Laboratory 8: Method appears very good.

Laboratory 9: Method considered to be very good.

Laboratory 10: Good procedure considering the determination. NH_4OH used instead of NaOH.

Laboratory 11: Versenate Method is more rapid and accurate than other specified procedures.

Laboratory 12: Endpoint determined with MonoVer indicator, which changes sharply from wine red to true blue when the last of the calcium and magnesium have been complexed by the standard TitraVer. We are quite satisfied with the method except in one respect. It appears that after a period of about 4-6 months, the MonoVer indicator becomes somewhat unstable, as is evidenced by a definite fading of the endpoints in our total hardness titrations from a true blue to a somewhat greyish green. In the past we have used Eriochrome black T, ManVer, and UniVer indicators and observed a similar degree of instability. Despite the difficulty however, we prefer this method to the use of the antiquated and most unsatisfactory standard soap solution. We should be pleased to learn whether any other laboratories have paralleled our experience with these indicators, or whether our experience is unique.

Laboratory 13: Endpoint is very sharp and the reproducibility excellent.

Laboratory 14: No comments made.

Laboratory 15: No comments made.

Laboratory 16: Precise replicate results obtained with this method. Endpoints very clear with either indicator except in cases with very high hardness, e.g. some boiler waters, where $CaCO_3$ may precipitate out, resulting in endpoint reversion.

Laboratory 17: Method rapid and gives satisfactory results.

Laboratory 18: No comments made.

Laboratory 19: No comments made.

Laboratory 20: No comments made.

MAGNESIUM

Reported as Mg. Amount Added = 85 ppm

Lab Number	Replicates	Average	Average Deviation Within Each Lab ppm	Average Deviation Within Each Lab per cent	Average Deviation from Amount Added ppm	Average Deviation from Amount Added per cent	Method
SEC	83 89 86	86	2	2.3	+1	+1	Gravimetric
1	85.9 86.5 86.0	86*	0.2	0.2	+1	+1	Gravimetric
2	86.3 86.9 86.3	86*	0.3	0.4	+1	+1	Gravimetric
	81*	81*	--	--	-4	-5	Calculated
3	95 96 92	94	1.6	1.7	+9	+11	Calculated
4	85 85 85	85	0	0	0	0	Calculated
5	90 91 90	90	0.4	0.4	+5	+6	Calculated
6	86.9 87.6 87.4	87*	0.3	0.3	+2	+2	Gravimetric
7	94 96 95	95	1	1.1	+10	+12	Calculated
8	73.4 74.4 71.9	73	0.9	1.2	-12	-14	Versenate
9	87 -- --	87	--	--	+2	+2	Calculated

10	83 84 84	84	0.4	0.5	-1	-1	Gravimetric
	92 100 100	97	0.4	0.4	+12	+14	Titan yellow
11	91.9 91.8 91.8	92*	0	0	+7	+8	Versenate
	88	88	--	--	+3	+4	Gravimetric
12	86.0 85.0 86.8	86*	0.6	0.7	+1	+1	Brilliant yellow dye (photometric)
	88.8 90.9 87.8	89*	1.2	1.3	+4	+5	Gravimetric
13	81 84 84	83	1.3	1.6	-2	-2	Calculated
14	87.8 88.0 88.0	88*	0.1	0.1	+3	+4	Gravimetric
15	85.1 84.7 85.6	85*	0.3	0.3	0	0	Gravimetric
16	89 88 89	89	0.4	0.5	+4	+5	Versenate (pontochrome TA)
17	88 88 88	88	0	0	+3	+4	Calculated
18	75 75 75	75	0	0	-10	-12	Titan yellow
19	89 -- --	89	--	--	+4	+5	Calculated
20	103 106 --	104	1.5	1.4	+19	+22	Gravimetric

* Denotes rounded off value.

COMMENTS ON MAGNESIUM DETERMINATION

Laboratory 1: No comments made.

Laboratory 2: Versenate method used only when quick Ca - Mg determination for private samples requested. Sufficiently accurate for all practical purposes.

Laboratory 3: No comments made.

Laboratory 4: No comments made.

Laboratory 5: We normally determine magnesium by subtracting the calcium value from total hardness. We have made a few determinations by precipitating the calcium as oxalate and then titrating the magnesium with comparable results.

Laboratory 6: No comments made.

Laboratory 7: No comments made.

Laboratory 8: Method appears very good.

Laboratory 9: Method considered satisfactory.

Laboratory 10: Precipitation and ignition time consuming and quantity of magnesium present is insufficient to give satisfactorily reproducible results in this lab. Quite satisfactory with Beckman D. U. spectrophotometer with photomultiplier attachment since most of our waters have less than 10 ppm. Fresh reagents are necessary and the procedure is somewhat involved.

Laboratory 11: Versenate method is regularly used. It is more rapid than the APHA method. The gravimetric method is time consuming and cumbersome.

Laboratory 12: In the photometric method, brilliant yellow dye is employed following the method suggested by Michael Taras in Analytical Chemistry, 20,1156 (1948). Carboxymethyl cellulose has been substituted as a stabilizing agent in place of starch or colloresin recommended by Taras. The advantages of the method are its ease of manipulation and speed of execution. The disadvantages appear to be an apparent degree of instability in the carboxymethyl cellulose and possibly in the brilliant yellow dye solution. As a result of this instability, it is necessary to determine a new curve of the optical density readings obtained on the spectrophotometer against known concentrations approximately every two weeks.

The alternative (gravimetric) procedure is in general used only when a check is desired on our photometric procedure, since we find the method time consuming and tedious. Nevertheless the method is an excellent one for determining magnesium, when particular emphasis is given to manipulation and technique.

Laboratory 13: Agreement with Standard Methods, 9th Edition (titan yellow colorimetric) is very satisfactory.

Laboratory 14: No comments made.

Laboratory 15: No comments made.

Laboratory 16: Method fine except occasionally, due to initial concentration of nondeterminate ions, an off-color (yellow grey) is observed.

Laboratory 17: Errors due to including other ions as magnesium are usually small enough to be neglected.

Laboratory 18: This method has proved very satisfactory for our purposes. Very little magnesium in waters of this State.

Laboratory 19: No comments made.

Laboratory 20: No comments made.

CHLORIDE

Reported as Cl. Amount Added = 195 ppm

Lab Number	Replicates	Average	Average Deviation within Each Lab ppm	Average Deviation within Each Lab per cent	Average Deviation From Amount Added ppm	Average Deviation From Amount Added per cent	Method
SEC	220 218 219	219	0.67	0.3	+24	+12	Mercuric nitrate
1	202 221 219	214	8	3.7	+19	+10	Mohr
2	220 220 220	220	0	0	+25	+13	Mohr
3	200 215 210	208	5.7	2.7	+13	+ 7	Mohr
4	220 215 215	217	2.2	1.0	+22	+11	Mohr
5	224 224 225	224	0.4	0.2	+29	+15	Mohr
	222 222 221	222	0.4	0.2	+27	+14	Potentiometric
6	209.6 209.6 209.6	210*	0	0	+15	+ 8	Mohr
7	223 224 226	224	1.1	0.5	+29	+15	Mohr
8	234 234 236	235	0.9	0.4	+40	+21	Mohr
9	219.6 218.4 218.4	219*	0.5	0.2	+24	+12	Mohr

10	219 219 219	219	0	0	+24	+12	Volhard
	215 215 214	215	0.4	0.2	+20	+10	Mercuric nitrate
11	233.0 232.9 232.9	233*	0	0	+38	+19	Mercuric nitrate
	232.3	232*	--	--	+37	+19	Mohr
12	223.0 223.0 224.0	223	0.6	0.3	+28	+14	Mohr
13	240 250 240	243	4.4	1.8	+48	+ 8	Mercuric nitrate
14	224.4 224.4 220.0	223*	2.0	0.9	+28	+14	Mohr
15	211.0 212.0 211.0	211*	0.4	0.2	+16	+25	Mohr
16	224 215 224	221	4.0	1.8	+26	+13	Mercuric nitrate
17	217 216 217	217	0.4	1.8	+22	+11	Mohr
18	253 255 255	254	0.9	0.4	+59	+30	Mohr
19	237 237 231	235	2.7	1.1	+40	+21	Mohr
20	225 225 --	225	0	0	+30	+15	Mohr

* Denotes rounded off value.

COMMENTS ON CHLORIDE DETERMINATION

Laboratory 1:	No comments made.
Laboratory 2:	No comments made.
Laboratory 3:	No comments made.
Laboratory 4:	No comments made.
Laboratory 5:	Mohr - Time and manipulation very good but precision on samples of low chloride is only fair. We don't adjust the pH for our routine samples.
	Potentiometric - Mercurous sulfate reference electrode with Ag-AgCl electrode and 0.05 ml HNO_3 very good with known samples, but can't be used for all routine samples. We have been unable to obtain satisfactory results with the mercuric nitrate method using diphenylcarbazone indicator, and are now investigating a potentiometric titration using mercuric nitrate.
Laboratory 6:	No comments made.
Laboratory 7:	No comments made.
Laboratory 8:	No comments made.
Laboratory 9:	Endpoint and matching of sample and blank considered difficult and subject to error.
Laboratory 10:	Volhard - Good procedure for high concentration, but not satisfactory for most of our waters due to low chloride concentration. Mercuric nitrate - Quite satisfactory, actually an improvement over the Volhard methods for quantities of chloride routinely found in our waters.

Laboratory 11: Mercuric nitrate - The method regularly used. Sharp endpoint is more certain than those specified in APHA.
Mohr - Difficulty encountered in obtaining a sharp endpoint.

Laboratory 12: Rapid; simple to perform; endpoint easily ascertained. Excellent method for our purposes.

Laboratory 13: We are set up for both the Mohr method and the mercuric nitrate method. Now using the mercuric nitrate method proposed by Taras in Analytical Chemistry, 22, 1020 (1950). The two methods check very closely and I prefer the sharper endpoint of Taras, and the fact that no titration blank is necessary. I do note that the purple complex formed is probably light sensitive (darkens on standing).

Laboratory 14: No comments made.

Laboratory 15: No comments made.

Laboratory 16: Very sensitive method if proper procedure is used, i.e., precise reduplication of standardized endpoint color, etc. Indicator must be refrigerated in order to insure long term stability.

Laboratory 17: Very satisfactory for normal concentrations. Concentration of very low chloride samples gives improved results for dilute waters.

Laboratory 18: No comments made.

Laboratory 19: No comments made.

Laboratory 20: No comments made.

SODIUM

Reported as Na. Amount Added = 25 ppm.

Lab. Number	Replicates	Average	Average Deviation Within Each Lab ppm	Average Deviation Within Each Lab per cent	Average Deviation from Amount Added ppm	Average Deviation from Amount Added per cent	Method
SEC	45 -- --	45	--	--	+20	+80	Flame Photometer
3	42.0 39.0 44.0	42*	1.8	4	+17	+68	Flame Photometer
4	40.0 41.0 40.0	40*	0.4	1	+15	+60	Flame Photometer
10	48 -- --	48	--	--	+23	+92	Flame Photometer
11	40 -- --	40	--	--	+15	+60	Flame Photometer
12	43.6 (Na and K reported as Na) --	44*	--	--	--	--	Calculated
13	36 36 36	36	0	0	+11	+44	Flame Photometer
16	38 39 38	38	0.4	1	+13	+52	Flame Photometer
17	42 42 41	42	0.4	1	+17	+68	Flame Photometer

POTASSIUM

Reported as K. Amount Added = 3.4 ppm.

Lab. Number	Replicates	Average	Average Deviation Within Each Lab ppm	Average Deviation Within Each Lab per cent	Average Deviation from Amount Added ppm	Average Deviation from Amount Added per cent	Method
3	8.5 9.0 7.0	8.2	0.8	10	+4.8	+141	Flame Photometer
4	8.5 9.0 8.5	8.7	0.2	2	+5.3	+156	Flame Photometer
10	3 -- --	3	--	--	-0.4	-12	Flame Photometer
11	3.4 -- --	3.4	--	--	0	0	Flame Photometer
16	3.4 3.4 3.5	3.4	0.03	0.9	0	0	Flame Photometer
17	3.7 3.7 3.6	3.7	0.03	0.8	+0.3	+9	Flame Photometer

* Denotes rounded off value.

COMMENTS ON SODIUM AND POTASSIUM DETERMINATION

Laboratory 3: The flame photometer was constructed in our shops and uses butane gas as fuel. The determinations were made using lithium as an internal standard.

Laboratory 4: No comments made.

Laboratory 10: Consistent results if routinely standardized. A fairly recent procedure for this lab.

Laboratory 11: Beckman Bul. 259, 28, (1951). Method is more rapid and believed to be more accurate than other specified directions.

Laboratory 12: Calculated as sodium, based on the concentrations of all the other anions and cations that have been determined. Determinations by ordinary methods are very difficult and tedious to perform.

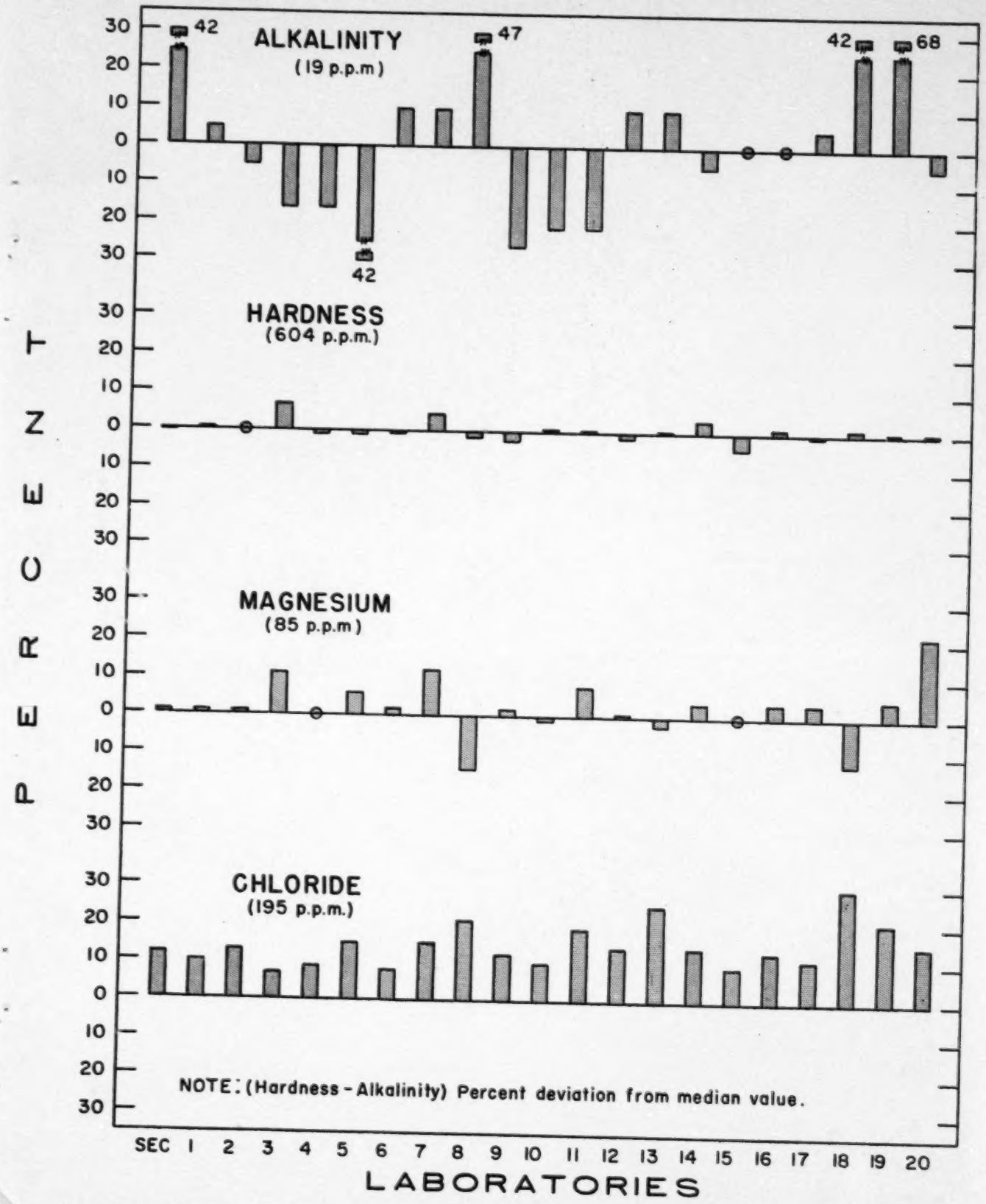
Laboratory 13: Have not found it necessary to use internal standards with the method, as recovery tests with representative samples have been good. Buffers have also been tried but did not give any appreciable increase in the precision.

Laboratory 16: Method requires only a fraction of the time for the extended method outlined in Standard Methods. Since it is a direct determination, it gives a "positive" result for the complete chemical analysis of water, rather than a determination by by difference in an anion-cation balance. This makes the balance more valuable since it affords a method of checking results of all determinations.

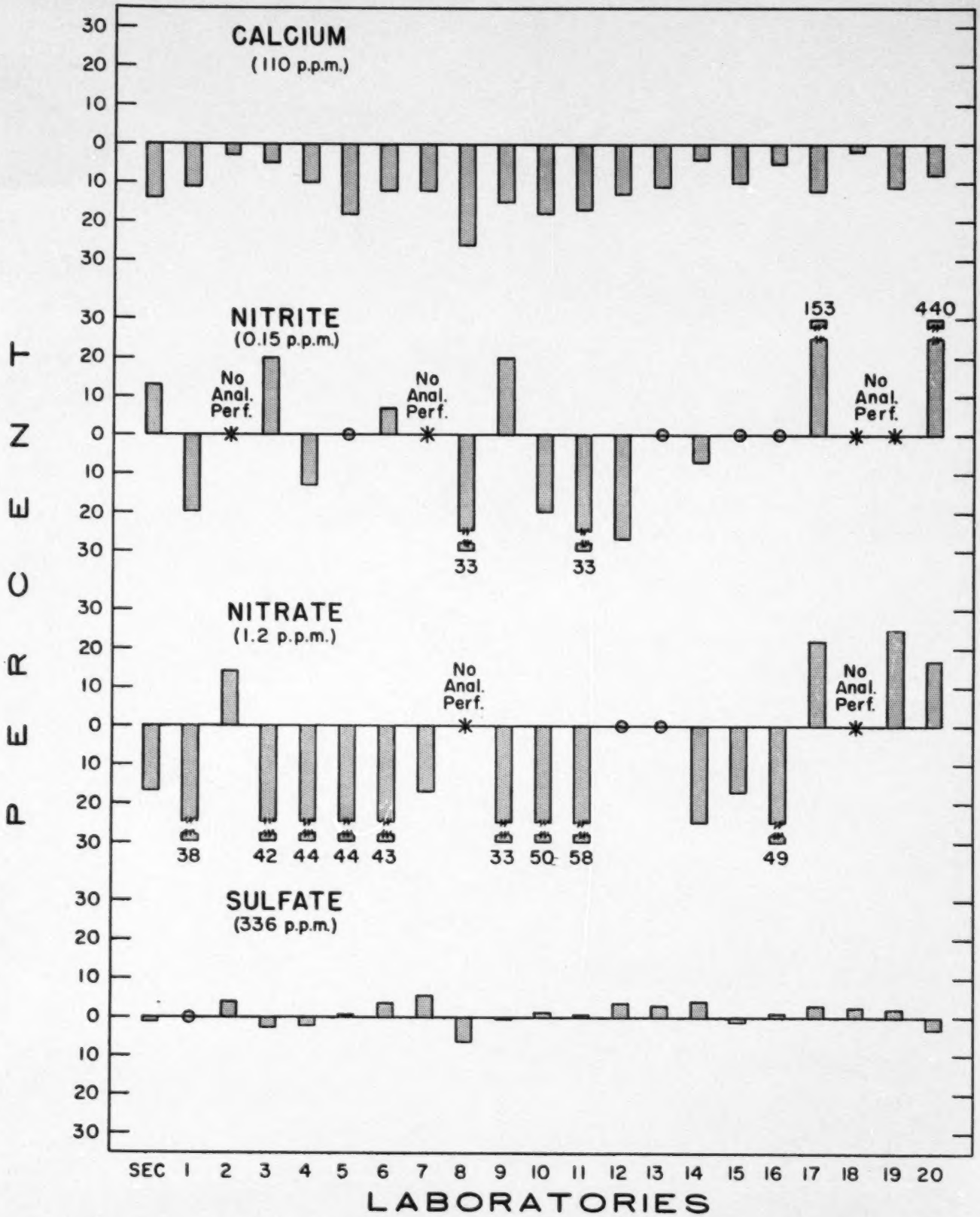
Laboratory 17: Standard 0-10 ppm curve is used to convert % T to ppm K. A maximum of 100 ppm Na is permitted in a sample without dilution. Standard curves of 0-25 and 25-100 ppm are used to convert % T to ppm Na.

SUMMARY OF RESULTS

Determination	Number of Labs Reporting	Arithmetic Mean ppm	Standard Deviation ppm	Median Value ppm	Average Per Cent Deviation Within a Lab	Average Per Cent Deviation From Amount Added
Nitrite	17	0.19	± 0.17	0.15	± 2.3	± 47
Nitrate	19	0.95	± 0.3	0.9	± 2.2	± 29
Sulfate	21	339	± 10	339	± 0.9	± 2.5
Alkalinity	21	20	± 5	19	± 1.0	--
Hardness	21	602	± 26	604	± 0.4	--
Calcium	21	98	± 7	98	± 0.8	± 11
Chloride	21	223	± 11	221	± 0.8	± 15
Magnesium	21	87	± 7	87	± 0.7	± 5.4
Sodium	8	41	± 4	41	± 0.7	± 55
Potassium	6	5.1	± 2.6	3.2	± 3.4	± 53



PERCENT DEVIATION FROM AMOUNTS ADDED



PERCENT DEVIATION FROM AMOUNTS ADDED